Supplementary information

Grain Size Effects on NiTi Shape Memory Alloy Fatigue Crack Growth

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Compact tension sample dimensions

After cold rolling to 1.00 mm (42 % thickness reduction), the compact tension samples were cut to the dimensions shown in Figure S1 with wire electrical discharge machining (EDM). After wire EDM cutting, the samples were heat treated for grain growth with the conditions from Table SI.

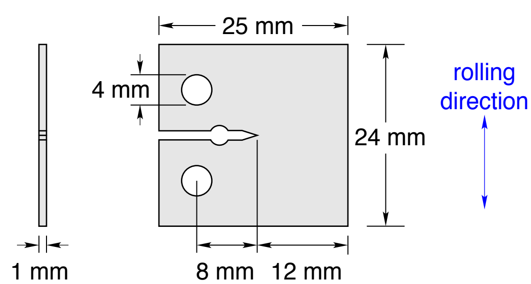

Figure S1: Compact tension samples conforming to ASTM E561 were cut with wire EDM from the cold-rolled sheets with the rolling direction perpendicular to the crack notch.

Table SI: Heat treatments for grain growth and the grain sizes measured by XRD and TEM.
∗ GS for the as-rolled condition was only measured with XRD.
+ The TEM field of view for the 1500 nm condition contained only a few grains (too few to make a distribution and statistics on grain size).

average GS, XRD (nm)	average GS, TEM (nm)	temperature (°C)	time (min)
10	n/a*	n/a	n/a
18	18 ± 12	250	45
42	48 ± 18	520	2
80	80 ± 13	520	6
1500	n/a+	600	45

Macroscopic experimental setup

The optical 3-D DIC system with electrodynamic fatigue system, described in Section 2.2 and shown in Figure S2.

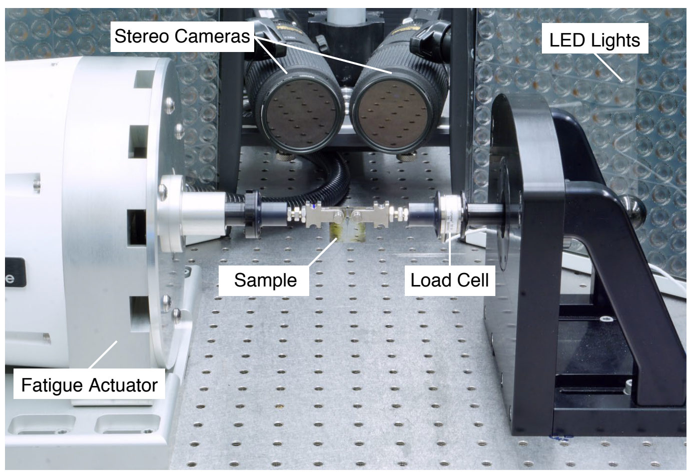

Figure S2: Macroscopic fatigue crack growth setup with optical 3-D DIC system, including cross polarization for enhanced optical DIC [1]. For a length scale reference, the grid spacing of the optical table breadboard was 1 inch.

High resolution TEM of 10 nm sample

The microstructure of the as-rolled, 10 nm condition shown in high resolution TEM (HRTEM) in Figure 2a and discussed in Section 3.1 is enlarged in Figure S3 for clarity.

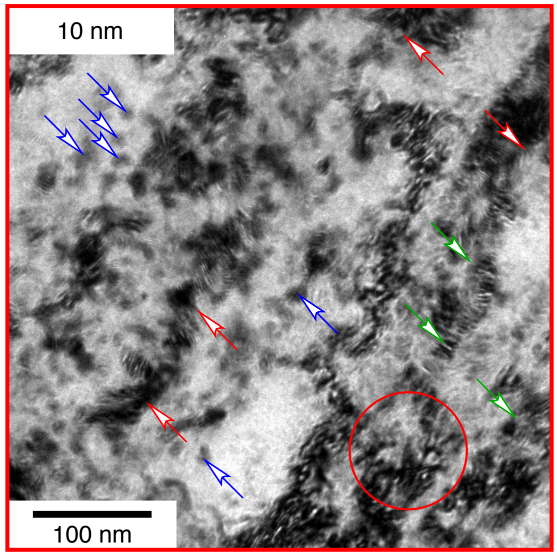

Figure S3: The HRTEM of the 10 nm condition shown in Figure 2a and discussed in Section 3.1 is enlarged in Figure S3 for clarity. The microstructure contains austenite nanocrystals (blue arrows) and twin lamellae of martensite (green arrows) with a high density of defects and lattice strains (red arrows). The red circle is the region for SAED from Figure 2a.

Microscopic observation of crack tip branching

At the microscopic level, crack branching and bifurcations occasionally created ambiguities in the crack tip location that prohibited valid relative crack displacement measurements. One such case is shown in Figure S4.

Graphical abstract

The graphical abstract accompanying the manuscript is provided in Figure S5.

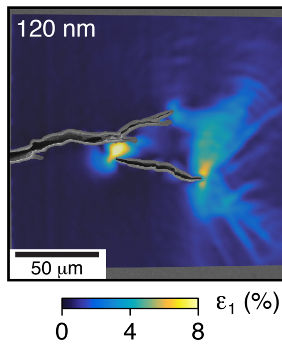

Figure S4: For a sample with 120 nm GS that was not included in the results of the main text, a significant degree of crack branching barred valid Δv measurements from the SEM-DIC displacements.

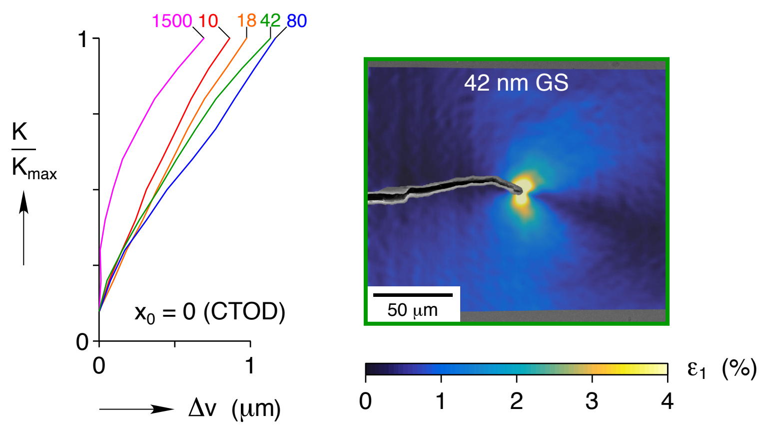

Figure S5: The crack tip opening displacement (CTOD) measurements at $\Delta v(0)$ indicate crack closure in the 1500 nm GS that was not present for the smaller GS. The major principal strain (ε_1) SEM-DIC field for the 42 nm sample exhibited a region of high strain ($\varepsilon_1 > 4$ %) about 10 µm away from the crack tip.

Supplementary references

1. W. LePage, S. Daly, and J. Shaw. Cross polarization for improved digital image correlation. *Experimental Mechanics*, **56**, 969, (2016).

2. P. Scherrer. Gottinger nachrichten math. *Phys*, **2**, 98 (1918).

3. S. Gao and S. Yi. Experimental study on the anisotropic behavior of textured NiTi pseudoelastic shape memory alloys. *Materials Science and Engineering A*, **362**, 107 (2003).